WHEAT FLOUR - ITS WEIGHT
AND MOISTURE CONTENT

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PREFACE

Many years ago the Association of Official Agricultural Chemists, an affiliation of Chemists connected with the various agricultural colleges and experiment stations, undertook to study and develop methods for the analysis of foods and of agricultural products. A method for determining moisture, seemingly a simple matter, was one of the first subjects to receive attention. One giving reasonably accurate, consistent and closely agreeing results was the goal, and while substantial progress has been made, a full measure of success has not been secured. Frequently the results have been conflicting and unexplainable, and analysts who have checked in other determinations have failed to agree on moisture tests. This failure has not been due altogether to lack of skill on the part of the chemist. Methods for the determination of moisture in many foods have never been fully perfected and seem to baffle all efforts for solution. The necessity for accurate moisture methods is well known and recognized by chemists. The Association from the beginning has been engaged in most valuable and necessary work, that has not been, at all times, as fully appreciated as it merits. Many of those who have taken a prominent part are no longer active, and their main reward has been the keen pleasure resulting from work well done. But although much has been accomplished, there is still vital work to be done.

For a number of years the writer of this article took part in the cooperative testing of methods of the A. O. A. C. and has served in various capacities in the organization, and for some years past has been engaged in the manufacture of flour. A part of the data in this article was presented in a brief prepared by request of the Bureau of Chemistry, U. S. Department of Agriculture, in June, 1922, and this more extended report is now made by request of the Millers' National Federation. In each case the report has been made without compensation. The brief and this more extended report have been prepared in the hope that by bringing together certain phases of the question, some solution of this much vexed problem may be suggested. A number of chemists and others have read the proof of this article and rendered valuable assistance.

Minneapolis, Minnesota.
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HARRY SNYDER

INTRODUCTION

The National Food and Drugs Act is a commendable type of constructive legislation and its faults, if any, are minor. The Secretary of Agriculture is designated as the chief executive officer for the enforcement of the act and of the authorized regulations which are made by the three Cabinet Secretaries. The Bureau of Chemistry is directed to make analyses and examinations of foods and drugs in accord with the methods prescribed by the Association of Official Agricultural Chemists, or by any method satisfactory to the Bureau. The three Secretaries of the Cabinet have issued 31 regulations (August, 1922) under authority of Section 3, and reserve the right to alter or amend the regulations at any time.

From time to time the Secretary of Agriculture issues service and regulatory announcements, and occasionally standards of purity for food products. While these Standards, as the Department of Agriculture states, do not have the force of law, they are issued "for the guidance of trade and regulatory authorities and represent what is recognized by best trade practices" as suitable standards.

In the enforcement of the Food and Drugs Act, certain practices and methods of procedure are followed. The Service and Regulatory Announcements, the Standards for the Purity of Food Products and the Methods of Analysis of the Association of Official Agricultural Chemists are simply agencies which the Secretary of Agriculture uses but they are not on the same legal basis as the Regulations made by the three cabinet officers.

Nearly all states have separate food and drug laws and sometimes they are not in harmony with the Federal laws and regulations. Then, too, some states have laws that parallel or even go further than the national law, in that all Standards of Purity of Foods and other regulations issued by the National Secretary of Agriculture are given the full force of law.

The miller does not object to reasonable rules and regulations but only wishes to know what they mean, so as to comply with them. Oftentimes he finds himself unable to understand the regulations, particularly where different states interpret them differently and where officials have different ideas as to the interpretation of the rules and regulations.

All laws, rules, regulations, standards and methods of analysis are bound in time to be subjected to rigid tests as to accuracy and they all must rest ultimately upon truth for their justification.

CONCLUSIONS

1. Five thousand cars of wheat tested by the U. S. Department of Agriculture gave moisture tests ranging from 7.4 to 22 per cent. The flour milling tests showed that good strong wheat must be tempered so as to carry up to 16% of moisture in order to be in proper condition for milling. With normal humidity conditions during milling, evaporation of water takes place so that the finished flour usually contains less than 13½% moisture, as determined by the water oven method of drying.

2. Tests conducted by the U. S. Department of Agriculture show that in the milling of wheat under good commercial practices, the flours contained from 12.12 to 13.32% moisture. This is equivalent to at least 13.12 and 14.32% moisture determined by the vacuum method of drying at 100° C and 27 inches vacuum.

3. Flour constantly changes in weight and moisture content according to the surrounding atmospheric conditions. It may dry to 9.00% moisture and less, or absorb moisture so as to test 15.00% or more; and the weight of the flour packages will vary accordingly.

4. Mechanical losses occur in handling flour that should not be considered due to short weighing of the packages or form the basis of a misbranding libel.

5. In a pile of flour the moisture content and weight of the individual packages may vary quite widely. In the same pile some bags may gain while others may lose moisture and weight.

6. Even in individual flour packages the moisture content is never evenly distributed, rendering sampling of flour for moisture tests extremely difficult. Different parts of a flour package may vary .75% in moisture.

7. In calculating dry matter content of flour packages, appreciable uncontrollable errors arise that must be taken into consideration.

8. The 13½% moisture standard for flour was published in 1904, about two years prior to the Food and Drugs Act. All commercial moisture tests of flour at that time were made according to water oven or air oven (100° C) drying methods. The U. S. Department of Agriculture made flour moisture tests according to these methods, and data used in formulating the 13½% moisture standard was obtained in this way. Also, the various official chemists used quite extensively in their work the water or air oven method of drying.

9. The Brown-Duvel method for the determination of moisture in wheat is based on water oven drying. The moisture content of wheat is taken into consideration in the federal grading of wheat, and extra dry strong wheat always commands a premium when sold on the market. In grinding dry wheat into flour the miller does not "get something for nothing," or make a profit from added water. Competition among the 7,000 or more commercial mills in the United States is very keen, particularly with little or no export business.

10. The present official method for determining the moisture content of flour is not accurate. The gluten proteins contain water chemically combined, or water of hydration, which is not free moisture but may be split off and included as such. Also mechanical losses, due to the finer particles of flour being removed, occur in the vacuum method of drying as the steady vacuum is maintained by the continual operation of a pump drawing a constant stream of air through the oven.

11. The records of the Proceedings of the Association of Official Agricultural Chemists do not show that drying foods in vacuo, as it first appeared in Bulletin 46 Revised, was ever considered or tested by the Association, or recommended by a referee either as a provisonal or official method.

12. The Bureau of Chemistry formerly held that when flour was packed full weight and without excess moisture and so long as it remained in the original package and was sound and wholesome, it complied fully with the Food and Drugs Act and subsequent deviations occasioned by natural agencies were not a violation of the act.

13. Weight and moisture content are joint attributes of flour. One cannot be considered without the other in weight adjustments and this is featured in Regulation 26.

14. Flour originally packed with less than 13½% moisture may absorb moisture and temporarily contain 15% and not be unsound; such flour should not be considered adulterated. The dry matter content of a flour package remains reasonably constant, but the difficulty is to control the factors which enter into its calculation.

15. For about 20 years the Association of Official Agricultural Chemists did not designate any specific method as official for determining moisture in flour and cereal products. There were official methods for other foods and feeding stuffs but none for flour, and drying in vacuo, as stated in Conclusion 11, was never adopted by

the association for determining moisture in foods and feeding stuffs as reported in Bulletin No. 46 Revised.

16. Chemists—official, industrial and commercial—get widely divergent results when testing the same sample of flour for moisture. Even when using the same method, the reults fail to check as closely as is desirable.

17. Many European flours with which American flours come in competition are milled with a high per cent of moisture. The American miller does not want a high moisture standard. He simply desires that the standard shall not be changed by changing the method of determining the moisture.

18. At the present time, there are inadvertently two flour moisture standards, one (the old standard) based on the water oven, and the other (the new standard) based on the vacuum oven method of drying. The official method of drying "in vacuo" for determining moisture in flour should be changed, or the 13½% water oven standard for flour should be adjusted so as to mean the same today as it did when adopted. When the conference was held with the millers at Washington in 1915, moisture testing of wheat and flour were considered on a parity (water oven basis).

19. The "Standards of Purity for Food Products" issued before the Food and Drugs Act was formulated and containing the 13½% flour moisture clause, were not authorized by congress for the guidance of food officials and courts as is frequently stated. The first appropriation, 1902, carried such a clause but in the next and last act, the 1903-05 appropriations, this clause was not enacted. Circular No. 10 contains the only standards issued under the first act. There is no standard for flour in Circular No. 10. Later standards promulgated in 1904-06, after Circular 10 was issued, were formulated under an act that did not carry any clause stating that the standards were made for the guidance of food officials and courts of justice.

20. Moisture absorbed or lost during the filling of packages, and uncontrollable deviations in manufacture due to sudden humidity changes should be considered as reasonable variations which attend the manufacture, filling and weighing of packages, conducted in compliance with good commercial practice. The operative miller must have a margin to cover such moisture changes so as to properly conduct his milling operations, as every 10% change in relative humidity, the U. S. Department of Agriculture finds, makes a difference of one half of a per cent in the moisture content of flour.

21. Some provision should be made whereby a miller can have his flour weighed by accredited weighmasters and marked accordingly so as not to be subject to libel actions because of uncontrollable atmospheric conditions.

22. Redrying and repacking sound flour packed full weight and with less than 13½% water, because it has absorbed moisture from the air, benefits no one except possibly those who are in the business of drying flour or those who may desire to get flour siezed in order to be released from contract losses arising from decline in the price of flour. Conditioning sound flour is contrary to ordinary commercial practices.

23. American flours tested in the open market containing up to 15% moisture have not been considered as adulterated by the Bureau of Chemistry and such flours have received the Bureau's highest commendation. "Owing to the firm attitude taken by American millers, the adulteration of staple brands of flour is practically unknown." Flours today contain no more moisture than when this statement was made.

24. The Millers' National Federation, the Association of Operative Millers and the American Association of Cereal Chemists have made certain recommendations relative to the moisture content and weight of flour: that the method for moisture shall include only the so called "free moisture" of flour, and in using the 13½ per cent moisture standard for weight adjustments it shall be on the basis of the moisture tests in vogue when the standard was formulated, the idea being that the standard shall retain its original meaning.

25. It is believed that these organizations named stand for the best trade practices on these questions and in no way would they countenance either the adulteration or the misbranding of flour or mill products as defined in the Food and Drugs Act.

American millers have almost invariably manufactured unadulterated flour and food products, dating from the time George Washington's flour, manufactured at Mount Vernon, was accepted in the West Indies and other ports without inspection. The same idea prevailed later when Abraham Lincoln worked in a flour mill and received the appellation of Honest Abe. Throughout the period of the Great World War the American millers, with the direction and aid of Herbert Hoover, were able to meet and solve the most complicated and colossal food problems the world has ever faced.

WHEAT FLOUR

Its Weight and Moisture Content

1. Hygroscopicity of Flour, and its Effect upon Weight.

Flour as the term is ordinarily understood is a product of wheat, and like wheat it is purchased and sold on a weight basis. A characteristic quality of flour is hygroscopicity or the power to absorb or discharge moisture according to circumstances. When flour is handled, stored or transported in ordinary commercial ways it may absorb moisture and increase in weight or it may dry out and lose weight, according to the surrounding atmospheric conditions. Deviations in weight and moisture content invariably occur, in fact flour could not remain fixed in weight and moisture content unless the atmospheric conditions as temperature, humidity, barometric pressure and rate of movement of air currents always remained constant. As hygroscopicity affects the weight and moisture content of flour it must be considered in commercial transactions and allowances must be made for its effect upon weight.

2. Water as a Component of Wheat and Flour.

Before considering the extent to which the weight and moisture content of flour are influenced by atmospheric conditions, due to evaporation or absorption of water, it is essential to consider water as a natural component of wheat and flour. Water, as hygroscopic or free moisture, is always present in wheat and flour. The extent to which it ordinarily occurs and the moisture requirements of wheat for milling as conducted in compliance with good commercial practice, have been determined by the U. S. Department of Agriculture.

The following statement relative to the "Moisture Requirements of Wheat for Milling Purposes," is copied from U. S. Department of Agriculture Bulletin No. 788 (page 2), a professional paper on Moisture in Wheat and Mill Products by J. H. Shollenberger, grain supervisor, in charge of milling investigations.

> "Wheat when received at the mill is seldom, if ever, in the best condition for milling, its moisture content being too high, too low, or not properly distributed throughout the kernel. To acquire the right moisture content for the outer and inner parts of the kernel, thereby insuring the best possible milling

condition for different wheats, requires the application of various methods of tempering. These methods may consist of a single, or successive, or of combined applications of water, heat, or steam, working through a period of time, ranging from a few minutes to as much as 36 hours, in order that the moisture may be properly distributed within the kernel. Dry climates and dry seasons naturally produce wheat of low moisture content, and damp climates and wet seasons produce wheat of high moisture content. There is, moreover, often considerable range in the moisture content of wheat during any given season and in any one locality. Moisture determinations of samples obtained from more than 5,000 cars of wheat, which were made by this department at Kansas City, Mo., during the years 1910 to 1914, inclusive, showed a range in the content of that factor from 7.4 per cent to 22 per cent. The fact that the moisture content of wheat may vary so greatly is evidence that the problem of properly tempering wheat is a complicated one."

To some who are not familiar with the process of flour milling it might seem that the miller deliberately adds moisture to his wheat so as to secure a greater weight of product. But a reasonable study of the facts precludes such an inference. The miller is well aware of the fact that excess moisture endangers the keeping qualities of his flour and would entail a monetary loss. When flour becomes unsound, invariably the miller must make good the loss, so he is not likely to add any excess moisture that would endanger the keeping quality his flour. For self-protection the miller wants to get away from all possibilities of unsoundness. In fact, the miller is more desirous of avoiding unsoundness of his flour than any one else, because in the end he is the one who must make good any losses. However, he must use water to condition his wheat for milling and this is, at times, a process difficult to regulate.

3. The Tempering of Wheat Is a Recognized Necessity and a Long Established Feature of Milling.

Any standard for moisture content of flour must necessarily recognize that wheat is tempered before it is milled, and that the strong wheats after they are brought into proper milling condition carry up to 16% or so of moisture, depending upon the character of the wheat to be milled. To mill hard wheats, the moisture content during tempering and conditioning is leveled up or down to about 15% (more or less). It makes no difference what the original moisture content of the wheat may have been, if it is above this point it must be lowered and if below, it must be raised.

When the miller buys his wheat the moisture content is considered. If it is extra dry, the wheat commands a premium compared to wheats with excess moisture. It cannot truthfully be said that the miller buys wheats of low moisture content, adds water in the milling process, and then sells the excess water in the flour, because the moisture content is taken into consideration in the grading and purchasing of wheat. The highest grade wheat is allowed 14% moisture (as tested by the Brown-Duvel method) and other grades proportionately more. The moisture standard for flour proclaimed by the Secretary of Agriculture many years ago is 13½%, maximum, water oven standard, which is also the method used for standardizing the "grain test".

At a comparatively recent date Food Officials have introduced a different method for determining moisture in flour (vacuum oven method) which secures a different per cent of moisture from the one used in establishing the flour standard. Unwittingly, there are two moisture standards: one based on water oven drying, and one based on the vacuum oven method of drying.

If the miller buys top grade wheat with 14% moisture, and makes this wheat into flour without gain or loss of moisture his flour tests 14% moisture according to the original water oven moisture standard, and 15% or so according to the vacuum standard used by many food officials. For some of the wheat grades, 15, 16 and 17% of moisture are allowed, and often the miller purchases wheat with 4% more moisture than is allowed in the flour. In a series of years the miller buys as much wheat that averages above 13½% moisture as below.

The lack of proper application and use of technical data from reliable sources have, no doubt, caused much misconception regarding this whole question of flour moisture. Fortunately, the U. S. Department of Agriculture now has its own data upon this question of moisture requirements for the milling of wheat and the resultant moisture content of flour, and Food Officials should make use of this data. A standard must rest upon exact experimental data. Recognizing the necessity for the use of water in the tempering of wheat in order to put it into proper condition for milling brings us to the question of how much moisture should be left in flour milled in compliance with good commercial practice.

4. Moisture Content of Flour Milled Under Good Commercial Conditions.

The bulletin quoted in a preceding paragraph (U. S. Dept. of Agr. No. 788) on Moisture in Wheat and Mill Products gives the moisture content of the wheat before and after tempering and of the various flour stocks resulting from the different reduction processes, and also the moisture of the final flours from three commercial mills in Kansas City.

	Per Cent of Moisture		
	Mill 1	Mill 2	Mill 3
Wheat before Milling	12.40	13.90	14.04
Wheat after cleaning and tempering	16.00	13.80	14.67
Patent Flour	12.12	12.97	13.32
Clear Flour	12.88	13.01	13.22

"In the case of the wheat samples, their moisture content was determined by use of the Brown-Duvel Moisture Tester, and in case of the other samples (flour, feed, and other mill products), the method used was that of drying to constant weight a small portion of the sample in a water bath oven, at the temperature of boiling water."

This method of drying flour to determine the moisture (as loss in weight) gives about 1% less water than the vacuum method of drying at a relatively higher temperature and under conditions which cause a greater loss in weight which is considered as moisture. The vacuum method of drying cereal products for determining moisture content is published in Methods of Analysis of the A. O. A. C., September, 1920, a book copyrighted by the Association (Price $5.00 per volume). In the preceding publication of these methods by the Department of Agriculture (Bulletin 107, Bureau of Chemistry), no methods are given for the analysis of cereals (see page 59, which is a blank page except for a statement that the methods are in preparation). During this interval and for several years preceding, there was no official method for determining flour moisture.

This statement concerning water oven and vacuum oven drying of flour and the difference in moisture results obtained by the two methods must be taken into consideration in dealing with flour moisture.

Had the vacuum method of drying, (extreme vacuum and high temperature) as followed by some food chemists been used in these tests the results would have been about 1% higher and approximately as follows:

	Per Cent of Moisture		
	Mill 1	Mill 2	Mill 3
Patent Flour	13.12	13.97	14.32
Clear Flour	13.88	14.01	14.22

All the flours except the patent of Mill 1 would, on the vacuum standard, have exceeded the 13½% moisture standard and been considered adulterated and illegal.

Many years ago the Division of Chemistry of the U. S. Department of Agriculture, made moisture tests of all the products of a flour mill. These early tests formed the basis of the manufacturing data for the formulation of the 13½ per cent moisture standard. The moisture was determined by drying in an air bath. These early tests and the later ones cited above, are on a reasonably comparable basis, as will be noted later.

There are certain features incident to the vacuum method of drying to be considered later which cause the results to be 1% higher than those obtained by water or air oven drying at 100° C.

For present purposes it is to be noted that in the tempering of wheat and the milling of flour under good commercial practice, as Bulletin No. 788 points out, from 12.12 to 13.32% moisture is left in the flour as determined by the water oven standard or about 13.12 to 14.32 by the vacuum standard.

5. Weight and Moisture Content of Flour Are Inseparable Attributes.

In dealing with flour, it is necessary to consider weight and moisture jointly, since a loss or gain of moisture causes a deviation in weight. To consider weight and moisture as separate and unrelated attributes is irrational and a violation of the laws of nature. A moisture variation due to unavoidable evaporation or to the absorption of water by packages in commerce, is a weight variation and is so considered in Regulation 26 (3)—Rules and Regulations—Food and Drugs Act. All standards must be applied in conformity to the principles of the Food and Drugs Act and the authorized regulations, and also be based upon good commercial practice.

A series of tests on the effect of the humidity of the air on the moisture content and weight of flour were made by the U. S. Department of Agriculture, Division of Chemistry about 1883. This early and carefully conducted work of Richardson seems to have been overlooked by many investigators, presumably because the bulletin is out of print and unavailable.

Richardson exposed at Washington, D. C., five lots of flour for 18 days and made, at intervals, fifteen moisture tests on each flour.

Tables are given showing the original moisture content, that is the moisture content at the beginning of the test, and "the weight 100 pounds of the original flour would have assumed under the conditions named" when exposed in a room, with free access to the air, "properly protected by a screen from exterior influences other than air."

A resume of some of the data follows:

March	Relative Humidity	Weight pounds	Patent Flours	Moisture content and weight at beginning of Test, March 7-8		Weight original 100 lbs., March 24th
7	.....	100				
8	46.4	100.65				
10	35.0	99.53				
11	59.0	101.73				
12	60.1	102.68				
13	34.0	99.88	Pillsbury A	100 lbs.	9.48%	102.88 lbs.
14	.....	101.08	Red River	100 lbs.	7.80%	104.87 lbs.
15	48.2	101.53	Frazee	100 lbs.	7.85%	105.20 lbs.
17	42.2	100.38	Pembina	100 lbs.	7.97%	105.95 lbs.
18	59.5	101.88	Minn.	100 lbs.	13.68%	99.35 lbs.
19	60.1	102.03				
20	55.6	102.48				
21	51.8	101.43				
22	51.1	101.68				
24	66.9	102.88				

The weight of the flour was dependent upon the relative humidity of the air. When the humidity was low the flour weighed less and with a relatively higher humidity the flour absorbed moisture and increased in weight. In these tests of the U. S. Department of Agriculture the weight and moisture content of flour are considered as inseparable attributes.

6. The Dry Matter Content of Flour.

The establishment of a moisture standard likewise establishes a dry matter standard, as dry matter is the product left after removal of the free or hygroscopic moisture. Theoretically, the dry matter of flour is reasonably constant and this is what should be considered in dealing with weight. As long as flour is sound and wholesome, the consumer is in no way defrauded if flour dries or absorbs moisture, provided the flour was originally packed full weight and without excess of moisture. On a dry matter basis such flours are neither

short in weight nor are they packed with excess moisture. When flour dries, the moisture removed must in turn be added in bread-making, and if the flour absorbs moisture from the air, then less water is used in bread-making. In either case, the same amount of bread, identical in dry matter content and nutritive value, is secured from the same weight of original flour.

7. Factors Affecting the Dry Matter Determination of Flour.

After flour enters Interstate Commerce it is the dry matter that must be taken as the basis of weight. Hence, it is important to consider carefully the factors which affect the calculation of the dry matter. A consideration of the basic principles upon which a dry matter calculation must rest includes:

1. Correct weight of the package.
2. Drawing of a fair representative sample for moisture tests.
3. Determination of the moisture by an accurate method.
4. The moisture test should be made by a skilled chemist.
5. In calculating back to dry matter, all mechanical losses of fine flour from packages during handling, transportation, and storage must be precluded.
6. Minor oxidation or respiration changes during storage must be considered.

Each of these factors may affect the accuracy of a dry matter calculation in flour.

8. Allowable Tolerances in Weighing Packages.

The Bureau of Standards of the U. S. Dept. of Commerce is the weight authority in this country. On platform scales used indoors for class A scales, a tolerance of two ounces per 100 pounds is allowed, or one-eighth of 1%. (Circular 61.) Granted that the scale used by a packer does not exceed the tolerance on beam for class A scales, a flour packer (that is the person who weighs and packs the flour) is liable to make either positive or negative errors in proportion to his skill and ability to correctly use the scale. Let us call this the human error. Regulation No. 26, Food and Drugs Act, allows for discrepancies in weight which occur in packing conducted in compliance with good commercial practice.

The U. S. Department of Agriculture has issued Bulletin No. 897 on weight variations of package foods, dealing with what are considered maximum errors of good commercial practice on single

packages, and on the average of a representative sample. On a 100 pound package the error in weighing is calculated as 5.65 ozs. for single packages and 2.92 ounces on the average of a representative sample. "If other than freshly packed goods are weighed, allowance must be made for shrinkage."

In arriving at these figures, the Bureau of Standard's tolerances on scales are used. Of the suggested 2.92 ounces deviation on one hundred pounds, average representative sample, 2 ounces are allowed for the scales error and .92 ounce for the human error in using the scales. It is proposed that the human error shall be less than half of the scales error allowed by the Bureau of Standards.

Regulation No. 26 says that in allowing tolerances for packing conducted in compliance with good commercial practice, the discrepancy shall be as often above as below the marked quantity.

U. S. Department of Agriculture Bulletin No. 897 (Weight Variations of Package Foods) states that the marked quantity, or the declared weight, must be used as the aim of the packer, and it suggests, as explained, tolerances for deviations covering single packages and for average representative packages; while Regulation 26 says the discrepancy shall be as often above as below the marked quantity.

Regulation No. 26 ought not to obliterate the tolerance allowed on scales as established by the Bureau of Standards, but the Regulation as interpreted by Food Officials does so. Regulation No. 26 would seem to mean: discrepancies are allowed for errors, such as human errors in weighing packages on scales which the Government, through its Bureau of Standards, declares are suitable scales (class A scales weighing 99 lbs. 15 oz., with an ounce tolerance either way, is a Class A Scales. That is, when the weigher eliminates all human error, he reaches 100 per cent accuracy, which is 99 lbs. 15 oz. on this particular scales. In practice, however, some food officials claim that when all packages are taken collectively the discrepancy must be as often above as below the marked quantity and the average be absolutely 100 pounds, omitting to consider that the weighings can be made only as accurately as the scales permit. When this unusual interpretation of Regulation 26 is made, then all tolerance allowed by the Bureau of Standards on scales are obliterated, which would not seem to be the intent of Regulation 26.

A reasonable interpretation must be made of the Rules and Regulations. There seems to be an idea among some food officials that only one weight tolerance is allowed on flour, that is (1) under Regu-

lation 26, and this tolerance is covered in Bulletin 897. The discrepancies under (3), due to evaporation or to absorption of water they think are included in the tables given in Bulletin 897, but this is not the case. It is to be noted that (1) reads "Discrepancies due exclusively to errors in weighing" etc., and (2) "Discrepancies in weight," that is, after a package is weighed and enters commerce. This (2) is confined to "differences in atmospheric conditions" in various places. Regulation 29 mentions two distinct tolerances. And there is no way these two tolerances can be considered as one and made a lump sum tolerance.

9. *Drawing Flour Samples for Moisture Tests.*

As flour is hygroscopic, great care must be taken in drawing the gross sample so that the three subdivisions forming the official samples will actually represent the flour in question. Samples drawn from the center and outer layer of a large flour package may vary in moisture content to the extent of .75%. Constant changes in atmospheric conditions from day to day are reflected in the weight and moisture content of flour packages. It takes time for all parts of the package to become uniform in moisture content.

No directions are given in the A. O. A. C. methods for the selection of samples of flour for purposes of analysis, as is done for many products, such as cheese, sugar, fertilizers, etc. When a uniform lot of flour is stored in a warehouse, some bags may gain in weight, while others lose, which makes it extremely important as to what bags are selected for sampling. The question of sampling is still further complicated by the fact that borings of dry flour from the center of a bag, exposed to a humid atmosphere, readily absorb moisture. Atmospheric conditions must (always) be considered when sampling flour.

Studies by Browne on the absorptive power of various carbohydrates show that starch is exceedingly hygroscopic. Starch makes up 70 per cent of flour. "While the absorptive power of the substances studied was highest in periods of high humidity and lowest in periods of low humidity, no fixed relationship could be established between rapidity of atmospheric fluctuations and the lag in the absorptive power of each material." (J. I. & E., Chem. Vol. 14, No. 8, p. 712.)

10. Are Present Methods for Determining Moisture in Flour Accurate?

Comparative moisture tests on the same sample of flour have been made by different chemists in co-operative testing work conducted by the Association of Official Agricultural Chemists, the American Association of Cereal Chemists and the American Institute of Baking. The results of these tests fail to show consistent agreement and it is a well known fact, often commented upon by chemists, that many of the present methods employed for the determination of moisture in flour fail to give accurate or consistent results. Even with the vacuum method of drying, Official Chemists report a ½% and more range in moisture content on the same samples when the flour is handled in glass containers so as to preclude any effects of humidity changes.

This lack of agreement of flour moisture results on the part of different chemists cannot be said to be due entirely to lack of skill of the chemist, but is largely due, as previously stated, to inherent defects in the method used. These defects are discussed in detail in another part of this report. As a matter of fact, as will be noted later, there is no record in the early proceedings of the A. O. A. C. that the vacuum method of drying foods for determining the loss of weight as moisture was ever adopted either as a provisional or an official method by the Association.

A regular progression of results are secured by the use of different, methods for determining moisture in flour. The different methods give reasonably closely agreeing results when always rigidly adhered to in the same laboratory and in the hands of individual chemists, "but agreement is not proof of accuracy."

11. Calculating Dry Matter Content from Weight and Moisture Percentage Does Not Give Original Dry Matter Weight As Packed Where Mechanical Losses Occur.

Whenever flour is handled, mechanical losses occur. The men who load cars, boats, or trucks are covered and coated with flour that shakes through the imperfections in the containers and through the needle holes that are made in sewing the bags. Rough handling increases the mechanical loss. This should be adjusted separately from short weighing of packages, because it is not short weighing. When mechanical losses occur the miller gains nothing. It is not

my purpose to argue that the purchaser of the flour should bear these losses, but the manufacturer should not be libeled when they occur, and packages that have sustained mechanical losses should not be used for calculating the dry matter of flour when packed. Ten or more needle holes the size of a pencil point made when the cloth was stitched and then stretched with the weight of the flour, will permit an appreciable loss during a long rough voyage. Salt in a shaker could not make such a trip without some loss, and flour is much finer than salt. Such losses may at times be attributed to excess moisture when packed, particularly if the package after sustaining such a loss were shipped into a humid region and absorbed moisture in excess of 13½%. Then, even if the flour failed to check in dry matter, the analyst would not be justified in testifying that the flour was manufactured with excess moisture.

In calculating the dry matter content of flour, a certain margin or tolerance is always necessary to cover these variable and human errors. Theoretically, the net weight of a package and the per cent of water contained in the flour should give the necessary data to calculate the dry matter content, but in actual practice the errors that occur appreciably affect the final results and a close agreement is not secured.

12. Calculated Dry Matter Content of Flours After Storage and Transportation Compared With Dry Matter Content of Flours When Packed.

The real test of the accuracy of the dry matter basis for judging as to whether or not a flour package was filled full weight must rest on carefully conducted tests. Such tests have been made by the U. S. Department of Agriculture on corn meal and to a lesser extent on flour. In the case of flour all of the tests and necessary data have not been published so as to be available for a careful review of the subject. The available data, however, suggest that appreciable mechanical losses occur and there are also, at times, certain chemical changes, such as respiration or oxidation processes that influence weight. In the warehousing of flour, ventilation is absolutely necessary, not only to remove excess of moisture, but other products as well, so as to conserve the baking value of the flour. Flour is not an inactive or chemically inert body when stored.

Briefly, a dry matter calculation of a flour package must rest upon an accurate weight on a standardized scale and a reliable moisture

test, made by a skilled chemist who tests a carefully drawn and what is known to be a representative sample, from which no mechanical losses have occurred. Unless a dry matter calculation rests upon such data, with reasonable tolerances, it cannot be accepted as accurate.

We come now to another phase of the moisture and weight question of flour.

13. When Flour Is Packed FULL Weight and Without Excess Moisture, Does It Not Comply With the Food and Drugs Act Provided the Flour Is Sound and Wholesome and Is In Its Original Container?

This question was fully discussed at a Hearing in Washington, D. C., June 5th and 7th, 1915, before the full Standards Committee, with Dr. Alsberg, Chief of the Bureau of Chemistry, presiding. There were present, representative millers, bakers, department solicitors and others. The record of the hearing, as taken by the Bureau of Chemistry, shows that a mutual understanding was reached.

> Dr. Alsberg: "The necessity for this discussion arises from a recent amendment to the Food and Drugs Act, which provides that a statement of the quantity of contents shall be stated in a plain and conspicuous manner upon a package of food in package form. Now what are we, as Officers entrusted with the enforcement of such a law, to do under the law with reference to the marking of quantity of contents with an article like flour, which by actual demonstration may run from 10 to 13½% moisture content?"
>
> Governor Lind: "That is a question that has been put to me professionally and I have said this: 'That every law must be reasonably construed.' The direct application of that law is to commodities or packages which inherently contain no factors changing the weight. That law should not be invoked or applied to a product which inherently, by reason of its chemical constituents, fluctuates in weight from time to time, provided it has been produced and packed under conditions which comply with the law at the time of its packing. Here they have complied with the law in manufacturing, packing, and marketing or sending into the channels of commerce—flour. The fact that flour may evaporate and change by reason of factors inherent in the commodity itself does not constitute a violation of the law. If you take the other position, there is no safety in a miller trying to do business a single day'."

Dr. Alsberg: "I think that is a reasonable way of looking at it and the law provides for tolerances—page 28."

Gov. Lind: "May I repeat in this connection a suggestion I made the other day, which in my experience is part of the construction or interpretation of every law? That (the law)[1] can apply only to commodities passing in interstate commerce that are invariable in point of weight. In respect of products variable in weight, the manufacturer's liability ceases when he delivers into the hands of the common carrier. That is a delivery under the law. When he delivers to the common carrier his commodity, that is a delivery to the consumer and if at that time it complies with the weight requirements, his full duty to the law and society is performed. Subsequent variations are of no concern to him. I am speaking solely from a lawyer's standpoint."

Dr. Alsberg: "We feel that way about it, Governor, and that is the reason why we have conducted a large investigation, which will take years to complete, concerning the normal shrinkage which takes place in shipping and transit, so that—"

Governor Lind: "You can reason back, yes."—page 126.

The record of this Hearing is unique in that the question of weight and moisture content of flour was discussed so thoroughly and by so many persons, all showing an earnest desire to reach a correct solution and a workable understanding of this question.

It would seem that the principles enunciated and agreed upon at this Hearing are logical and sound, and should prevail. They are the views alike of the Department and of millers and represent the best trade customs and practices relative to this question of weight and moisture content of flour.

A careful study of the records of the Hearing show that there was a general agreement of opinion that it was best not to make any change in the 13½% moisture standard which, as Dr. Alsberg explained, was to be used for weight regulatory purposes. At that time there was no official method for the determination of moisture in flour, and the discussion must have been based on the methods then in vogue.

14. What Was the Prevailing Method or Methods in Use for Determining Moisture at the Time of this Hearing?

At that time the Brown-Duvel method for determining the moisture content of grains had been developed by the U. S. Department of

[1] Added by writer.

Agriculture and had been adopted and used by the trade generally. This method is standardized against what is known as the water oven method of drying; that is, the loss of weight which occurs when the grain is dried in a water oven heated to the temperature of boiling water. The loss of weight in drying is called the water or moisture content.

At the Hearing, the moisture in the wheat and in the finished flour were spoken of interchangeably. The millers, and members of the Standards Committee and all who discussed the question spoke of both the moisture in the wheat and in the flour often in the same sentence, plainly showing that they were used on an equality basis, and that one was strictly comparable with the other. Furthermore, drying in a water oven and in an air oven at 100° C. or so were the only methods in common use at that time and they were the only methods with which the trade was familiar.

As the Brown-Duvel method was used at that time for grading wheat, it is inconceivable that the use of another method for determining moisture in flour, particularly one standardized on a different basis and yielding different results could have been entertained by the millers at this hearing. Wheat is 75 to 100% flour depending upon the kind and quality manufactured and the moisture of the flour, in or out of the wheat, should be determined by the same standard. A double moisture standard for wheat and flour is not commercially sound.

15. The Methods for Determining Moisture in Wheat and Flour Should Be Retained on a Parity.

The necessity of having wheat and flour retained on a moisture parity is so axiomatic that it would seem unnecessary to discuss the subject. The Grain Standards Act provides for the establishment of the method for determining moisture in wheat. The Brown-Duvel method, as described in Circular 72 (Bureau of Plant Industry) is the official method. The moisture can also be determined "or ascertained by any device and method giving equivalent results" (Sec. 4). Much care seems to have been taken in standardizing the method so as to secure the free moisture present in the grain, as in some cases the drying of the whole grain was continued for several days.

This method of drying comes much nearer giving the actual moisture content than the severe vacuum drying method followed

by some food officials. The Brown-Duvel method is standardized on the same basis as the original 13½% moisture standard. Methods developed or modified at a later date, giving higher results, have virtually the effect of changing the moisture standard. Whenever the method for determining the moisture content is changed, then the standard should be changed to correspond.

16. The American Association of Cereal Chemists' Moisture Method.

The American Association of Cereal Chemists adopted about 3 years ago a method which is, in brief, drying in an air oven for 4 or 5 hours at about 103° C. This method is followed by many chemists and, with minor modifications, is used in many official laboratories, but gives different results from the A. O. A. C. vacuum oven method.

17. Defects of the Present Vacuum Oven Method for the Determination of Moisture in Flour.

The present official method for determining the moisture content of flour reads: "Dry a quantity of the substance, representing about 2 grams of dry material, in a current of dry hydrogen, or in vacuo at the temperature of boiling water to constant weight (approximately 5 hours). If the substance be contained in a glass vessel, the latter should not be in contact with the boiling water." This is interpreted by some chemists to mean a temperature of 100° C, a vacuum of 27 inches, and a prolonged period of heating. Water boils in a vacuum oven at about 57° C. when the vacuum is 25 inches. When the oven temperature is raised to 100° C., there is an excess of 43° C. over the boiling point of water. Under such conditions, a greater loss occurs than when the flour is heated at the same temperaturr in an air or water oven.

Heating in a vacuum oven for five hours, coupled with the strong suction, may introduce three sources of error:

(1) Removal of chemically combined water from the proteins, gliadin and glutenin.

(2) Mechanical losses.

(3) Minor losses from dissociation of other components of the flour.

18. Water of Hydration of the Wheat Proteins.

The presence of water in flour chemically combined as water of hydration is determined and reported in U. S. Department of Agriculture, Bulletin 101, O. E. S.

"*Water of hydration of wheat proteids.* As previously stated protein is calculated in these investigations by the factor 5.7. But the product represents strictly anhydrous proteids, while the proteids present in flour are not in that condition. When dried at 100° C. the gliaden retained 4.22% water, which was expelled between 101° C. and 102° C. and the glutenin retained 4.66% water which was likewise driven off only with difficulty. It is difficult to separate and obtain these proteids in a pure form without chemical changes. From these facts and from results obtained with the bomb calorimeter, as explained below, it would appear that as ordinarily present in flour the gluten proteids are in fact in hydrated forms.

"The gluten proteids, gliadin and glutenin were prepared from the gluten obtained in the separation of starch. The gluten was cut into small pieces and extracted for several days with 70% alcohol. The gliadin obtained from the alcohol extract was redissolved, purified, and dried over sulphuric acid. Both the gliadin and glutenin hold water very tenaciously and can be dehydrated only with difficulty. The gliadin contained .34% ash and the glutenin .44% which were taken into account in determining the heat of combustion." The heat of combustion for gliadin is 5924 and for glutenin 5879 per gram.

Osborne in "The Proteids of the Wheat Kernel," states that after purification with absolute alcohol, and ether he dried his proteins at 110° for analysis after he had first thoroughly dried them over sulphuric acid, and in the case of gliadin, the loss in drying was equivalent to 9.2% water. He speaks of gliadin as not suffering any change in the separation and preparation for analysis.

The possibility of the addition of water to the proteins during extraction and of the water being held physically are precluded in both Osborne's and Snyder's results. In both cases the proteins were purified with absolute alcohol and ether and these reagents would have removed any physically bound water. The 4.22% water found by Snyder expelled at 103° C. and the 9.2% found by Osborne expelled at 110° C. could come from no other source than the water chemically bound with the protein.

After once passing 110 to 120° C. in an evacuated system, this being the temperature beyond which water chemically combined with the proteins is given off, Nelson & Hulett find (J. I. & Eng. C. Vol. 12 No. 1) flour can be heated, out of contact with the air, to a comparatively high temperature before the point is reached where it

gives off appreciable amounts of gaseous products. It is not unreasonable to expect, after both the free and chemically combined water are expelled, that a period of fairly stable equilibrium may follow, when flour is heated out of contact with the air.

19 Mechanical Losses in Vacuum Drying.

When the vacuum pump creates the vacuum, air is pumped out of the oven. To maintain the vacuum, the pump must be kept in constant action. The suction created removes fine flour particles and these are counted as moisture losses. That such losses do occur is easily demonstrated by passing the air as it leaves the oven through a wash bottle containing distilled water. The wash bottle, acting as a "dust collector," is placed between the oven and the pump. The solution becomes cloudy and suggests milkiness. The microscope shows flour particles in suspension. The amount of loss as flour dust is difficult to determine as part sticks in the tubes and dries like gum.

The writer of this article, in co-operation with Dr. Frankforter of the University of Minnesota, has undertaken a study of the subject of vacuum and other methods of drying. Our preliminary work shows that it was necessary to make a special copper lining for the vacuum oven to prevent flour particles lodging on the rough interior walls. The mechanical losses are variable; at times they are small, and then unexpectedly they are appreciably more and if the test is prolonged they may make up .20% of a moisture test. Coarsely granulated flour products, as grits, suffer less loss than the finest granulated flour.

The main loss or error introduced in the extreme vacuum drying of flour arises from the dislocation of the combined water or water of hydration of the proteins. Applying Osborne's data, a flour would yield an appreciable amount of water chemically combined with the protein.

Mechanical losses, as flour particles sucked out by the vacuum pump, as in vacuum cleaning, and water of hydration cannot be considered as the hygroscopic or free moisture content of flour.

The use of strong vacuum and 100°C for drying flour give results that are inaccurate and too high and is an unwarranted interpretation of the method. Vacuum drying was introduced with the idea of securing drying at a low temperature so as not to induce chemical changes in the substances dried.

The argument that the high vacuum drying of flour because it secures maximum results, must be accurate, has no foundation.

This data, so far as the defects of the vacuum oven method of drying are concerned, is introduced mainly to show why vacuum oven drying gives higher results than water oven drying, and why the U. S. Department of Agriculture, Bureau of Markets, in developing a method for determining moisture in wheat and other grains gave preference to the water oven method.

The main question: What the 13½ per cent moisture standard really means, is concerned only with the method employed in determining the moisture at the time the standard was formulated. As will be noted in the following section, the standard was formulated on the water oven drying method and not on the vacuum oven method. At that time water oven drying was the method in use in ordinary commercial practice. All standards must ultimately rest upon the best commercial practices.

20. The Thirteen and a Half Per Cent Moisture Standard for Flour is Based on the Water Oven Drying Method.

The Standards Committee reported a definition for flour to the Secretary of Agriculture, December 19th, 1904, nearly two years before the enactment of the Food and Drugs Act:

"Flour is the fine, sound product made by bolting wheat meal and contains not more than thirteen and one-half (13½) per cent of moisture, not less than one and twenty-five hundredths (1.25) per cent of nitrogen, not more than one (1.0) per cent of ash, and not more than fifty hundredths (0.50) per cent of fiber." (Bureau of Chemistry, Bul. 69 (Revised) Part 1.)

The data upon which this standard rests is published in Bulletin No. 13, Part IX. The Prefatory Note to this Bulletin says: "The examinations of flours had for their primary purpose the establishment of a standard of composition. These analyses were made chiefly in the years 1894 and 1895." They were made consecutively with analyses mentioned in Division of Chemistry Bulletin No. 45, issued April 15, 1895. This bulletin states: "Determination of Moisture. Two grams of the substance in a flat bottomed aluminum dish are dried for five hours at the temperature of boiling water. Experience has shown that after this time no further loss of weight takes place." The analyses of the flours mentioned on page 7, Bulletin No. 45, are all published for the first time along with other

flours, in Bulletin No. 13, Part IX. These two bulletins are inseparable as far as the testing of flours is concerned. While no specific mention is made of the moisture method in Bulletin No. 13, the invariable rule is that when a connected series of tests are made, as in this case, the method of testing is described in the first publication, and it is not changed in subsequent publications without recording that fact. As the flour work in Bulletin No. 13 is a continuation of Bulletin No. 45, published in 1895 and at the time the last flour work in Bulletin No. 13 was done, it is only necessary to refer to Bulletin No. 45 to determine the moisture method employed in this work. The water oven method that was followed is given in this paragraph.

Bulletin No. 13 also gives eighty-eight "Analyses of Products of Roller Milling," made by Clifford Richardson of the Department in 1883-84. At that time a change was just beginning to be made from the mill stone to the roller system of milling. The samples were drawn from Pillsbury A Mill. Richardson's moisture method, described in a preceding bulletin was: Flour "One gram was dried in a porcelain crucible at 100°-105° C until it ceased to lose weight." Richardson fully realized that flour manufactured by the mill stone method was different from the product of the modern roller mill. He mentions but does not use the earlier work as Brewer's, reported in the tenth U. S. census (1880) and based largely on Kedzies' analyses of mill stone flours in 1877. Such data could not be used in formulating a standard without violation of Principle 7 governing standards. "Standards are based upon data representing materials produced under American conditions and manufactured by American processes, etc." The "stone age" of milling does not represent modern roller process milling. Richardson's analyses are not included in the final averages of bulletin No. 13.

In addition to the 88 analyses of products of roller milling by Richardson, and the 41 samples exhibited at the World's Columbian Exposition, 81 samples of flour, including "flap-jack" and pancake flours are reported in Bulletin No. 13.

The water oven method for determining moisture was used in all of the new work upon flours published in Bulletin No. 13, where "The examination of the flours had for their primary purpose the establishment of a standard of composition." The reason for using this method is stated in Bulletin No. 45 page 11. "Experience has shown that there is practically no difference in the analytical data

secured in samples dried in the open air in a partial vacuum, and in a current of hydrogen, and for this reason the drying in the air, which is so much more easily accomplished, has been followed."

It is evident that if any other methods than water oven drying had been used by the Bureau of Chemistry at this time they would have been operated in such a way as to give the same results as the water oven method.

The average moisture content of the flours of Class I, reported in Bulletin No. 13, Part IX, was 12.77 equivalent to 13.77 per cent or more on the basis of vacuum oven drying at 100° C and 25 inches of vacuum. "The samples whose analyses are given under Class I may be regarded as representing the best high-grade patent wheat flour on our markets." See page 34 of this article.

Not only is the method used for determining the moisture described but the analyst who did the work used air drying many years later on products more susceptible to changes by air drying than flour, namely fruits and fruit juices (See Section 26).

Open air drying was recommended and favored by the Bureau of Chemistry at that time. Wiley in his "Principles and Practices of Agricultural Analysis," Vol. 3, 1897, discusses the advantages arising from drying in open air at the temperature of boiling water on page 34, and "there is left for the worker in the laboratory the choice of processes already described, etc." p. 35. All of the data and records show that "the choice of processes" was "drying in the open air at a temperature not exceeding that of boiling water."

This is not a question as to the relative merits of analytical methods for determining moisture in flour, it is simply a question as to the methods used in testing the flours that were considered in formulating the 13½ per cent moisture standard in 1904. It will be noted later that water oven drying gives lower results than vacuum oven drying 100° C and 25 inches vacuum. See Sections 28, 30 and 31.

The relative merits of white and whole wheat flours are noted by Richardson who quotes Rubner's work that white flour is more digestible and cheaper weight for weight than whole wheat flour:

> "We can only hope, then, for an improvement in the character of our wheats to add to their nitrogen content, and to improved methods of milling, which we are fast becoming possessed of, to make it possible to produce flour with the highest amount of nitrogen in the higher grades, and at the same time with it the best physical condition. Then we may expect to improve our breads."

21. Drying Foods in Vacuo at 100°C and 25 inches Vacuum for the Determination of Moisture was never adopted as an Official Method.

The present official method, as previously stated, first appeared as official for cereals in the A. O. A. C. Methods, published in 1920. The preceding publication of the Methods by the Government in 1908 (Bulletin 107) gave no methods for cereals.

The methods for 1920 were read and all new methods were acted upon by the Association. Any claim that the vacuum method of drying (with heat) is official rests upon its having been published as official in Bulletin 107 "V General methods for the Analysis of Foods and Feeding Stuffs," page 38.

As early as 1899 (See Bulletin 56—page 129) a division of Cereal Products was created entirely separate from the division of Cattle Feeds and special methods for their testing were contemplated.

See also Provisional Methods A. O. A. C. for the Analysis of Foods, Bulletin 65 Bureau of Chemistry, page 41.

IV.—Cereal Products

by A. McGill

Chemist of Inland Revenue Lab. Ottawa, Can.

> "It has been found impossible to prepare the report on this subject this year. The heading has been inserted here to preserve its proper order."

It was generally recognized by the Association that there were no official methods for testing cereals, after this subdivision had been created. See Proceeding A. O. A. C. 1910, Bulletin 137, page 119 "Cereal Products."

> "It is recommended—
>
> (1) That the associate referee on cereal products be instructed to devote special attention to methods for analyzing and testing wheat and flour.
>
> Carried. (Included in supplementary report of committee C. Attention was called to the fact that no methods for cereal products are given in Bulletin No. 107, Revised, and that milling and baking tests were also needed.)"
>
> "Water in Foods."
>
> "It is recommended—
>
> (1) That the vacuum method for the determination of moisture in foods be further studied, etc."

Numerous other references could be given showing that the association recognized that there were "no methods for the analysis of cereal products."

Referee White in his report to the A. O. A. C. for 1911 recommended:

1. "That the method for the determination of moisture in Bulletin 107, revised, page 38 (1), be made official for cereals." (Also vacuum dessicator method was recommended for further study.)

For purposes of comparison, this method and the method of 1920 are both given. The precaution as to drying, common to each method is omitted.

From Bulletin 107 Revised 1908.

"Dry a convenient quantity of the substance representing about 2 grams of dry material, at the temperature of boiling water until it ceases to lose weight (approximately five hours), in a current of dry hydrogen or in vacuo."

This method was official for Foods and Feeding Stuffs, but did not include flour.

From Method of Analysis A. O. A. C. 1920.

"Dry a quantity of the substance, representing about 2 grams of dry material, in a current of dry hydrogen or in vacuo at the temperature of boiling water to constant weight (approximately five hours).

This method is now official for Foods (including flour) and Feeding Stuffs.

The "in vacuo" moisture test for flour and cereal products now becomes linked with the method for Foods and Feeding Stuffs, and its claim as official for flour rests with the original action taken in regard to this method.

Drying in vacuo, printed in the Official Methods, first appears in Bulletin No. 46, Revised Edition 1898, page 23: "Dry from 2 to 3 grams of the substance for five hours, at the temperature of boiling water, in a current of dry hydrogen or in vacuo." In the first edition of bulletin No. 46, published in 1895, (not the revised edition,) the sentence ends with hydrogen.

Therefore the status of drying "in vacuo" rests upon whatever action was taken by the A. O. A. C. between 1895 and 1898, as drying "in vacuo" does not appear in the 1895 but does appear in 1898 methods.

Let us first consider how a method becomes "official." To become official a method must be subjected to the following tests, according

to the preface in "Methods of Analysis A. O. A. C.," published in 1920.

> "To attain the aims of the association for a set of accurate methods, a system was evolved by which the methods in question are subjected to the most rigorous and painstaking scrutiny before they can be adopted. A "referee" is appointed for any subject for which the association has not yet an official method or for a method which seems to require further investigation. The referee conducts analyses according to the methods suggested for adoption in comparison with methods already established, obtaining the collaboration of as many as possible of the workers in that field. In addition, a great deal of original research has been inaugurated on new methods. This system developed logically until at the present time, in order to be adopted as "tentative," a method must be recommended to the association by the referee, and such recommendation is made only after the method has undergone a thorough collaborative and critical study. Further, the special committee on methods must approve the recommendation and the method must be accepted by a vote of the association. In order to become "official," a method must be again accepted at another annual meeting. The recommendations of referees are published in the reports of the proceedings of the association in the *Journal of the Association of Official Agricultural Chemists*, so that all tentative methods are made public before being adopted. This permits consideration and criticism by chemists who are not members of the association. It is immediately apparent that a method can be made official only after the most thorough series of tests, not alone for accuracy, but for ease of operation as well. It may be stated without reservation that more elaborate and painstaking effort has been expended on this collection of analytical methods than upon any other set of similar methods in the field of chemical science."
>
> This Preface is signed by C. L. Alsberg, Secretary of the Association of Official Agricultural Chemists.

Did this "or in vacuo" method follow the usual channel of adoption?

How and when was drying in vacuo made official?

No mention is made in the Proceedings of the Association of Official Agricultural Chemists for the years 1895, 1896, 1897 or 1898 of the vacuum method of drying having been tried, proposed as a provisional method, or finally adopted as an official method.

I have all of these reports. I have examined them carefully, and so have other chemists; the only way in which vacuum drying is mentioned is in connection with Carr and Sanborn's paper (1895)

page 134 on "The Dehydration of Viscous Organic Liquids." In the discussion following the paper Dr. Wiley aptly states: page 152.

> "I would like to suggest the importance of drying at low temperatures. In drying levulose at the temperature of boiling water in the open air, there is a progressive decomposition of the substance, so that the apparent amount of water obtained is much greater than the real percentage of water present. In the drying of honeys, low-grade sugars, and molasses of all kinds, the error is a very important one. It seems to me that the official method should be changed when the proper time comes to include possibilities of that kind. It is easy to dry in a partial vacuum, and it seems to me that all organic liquids, whether viscous or not, and all organic bodies for chemical examination or for the determination of moisture should be dried in partial vacuum. There would be no danger of oxidation by the proposed method; the drying could be accomplished more regularly and with less danger of injury to organic substances. Better results could be obtained by adopting a method which would include the good points of all."

In the 1896 report, Lindsey,, referee on Cattle Feeds, mentions that he had discontinued "comparisons of results obtained by different analysts in determinations of moisture, crude ash, fiber, fat and protein. He believes these methods to be as perfect as they ever can be made, XX." Page 46. In the 1897 report no comparative tests for moisture by vacuo are given nor is there any indication that any such studies or tests were made. Provision is made for republication of methods, on page 153. There are no recommendations for any changes in moisture determinations in Foods (then known as Cattle Feeds).

In the 1898 report Krug, referee on Cattle Feeds, gives the moisture content of the three samples used in the estimation of starch. These results are only incidental and no mention is made of methods employed. It is stated on page 88 that the Carr and Sanborn method had been provisionally adopted by the Association for water in sugar and molasses, but had not received much attention from the association chemists.

The Methods of Analysis A. O. A. C. bulletin 46 (revised), bears this legend on the title page:

> "Adopted by the Association of Official Agricultural Chemists November 11, 12 and 14, 1898."

There is no record in the 1898 Proceedings that these Methods of Analysis were ever reported to, received, discussed, adopted or acted upon by the Association.

It is in the 1898 methods that vacuo first appears printed in the official methods.

The records of the Association of Official Agricultural Chemists do not show that drying foods in vacuo with heat as applied to the division of Foods and Feeding Stuffs was ever even considered or tested by the Association or recommended as a provisional method or finally adopted as an official method.

Had the method been tested first as provisional like other official methods, its failure to give results would have been observed, and the time, temperature, and vacuum conditions would have been studied.

22. Drying in Vacuum Without Heat.

Method No. 2, for moisture in Foods and Feeding Stuffs, official, page 71 (Methods of Analysis A. O. A. C) was developed by Dr. Trowbridge who recommended it for general use in the analysis of foods, except factory control when rapidity is necessary. Dr. Trowbridge states: "Most food products undergo more or less change upon being heated for several hours at 105° C even if it is done in a vacuum or in an atmosphere of hydrogen." Page 150, 1909 A. O. A. C. Proceedings. This method was regularly adopted by the Association.

High temperature vacuum drying was not the original intent when vacuum drying was introduced. The whole idea was to avoid high temperature, and to get the moisture without heating to 100° C. When "or in vacuo" was placed on the end of the sentence, 1895 Methods, it meant that vacuum drying was a new method and it was separate and distinct from hydrogen drying and the conditions different from those governing hydrogen drying. It was first drying in hydrogen, if the analyst elected to do so, or if he did not dry in hydrogen he could dry "in vacuo" without temperature or pressure restrictions. Without these limitations chemists have failed to get concordant results.

23. To What Extent do Flours Range in Moisture Content?

A flour that is manufactured with not to exceed 13½ per cent moisture and packed full weight may gain or lose in weight and moisture

content according to the prevailing atmospheric conditions where the flour is stored. In Bulletin No. 13, previously mentioned, the moisture content of the flours ranged from 9.39 to 15.30 per cent for the first class of flours, and for other classes 9.28 to 15.71 without being considered adulterated, as extracts from the bulletin show:

"*Discussion of Results—Class 1.*

"The samples whose analyses are given under Class 1 may be regarded as representing the best high-grade patent wheat flour on our markets." Page 1257.

"*Moisture.*

"In the samples of Class 1 there is an extremely uniform percentage of moisture. The variations from the mean, which is 12.77 are usually very small. The extreme plus variation is found in sample No. 12549 with a content of moisture of 15.30 per cent, the variation being 2.52 per cent in this case. The minimum percentage of water is found in sample No. 12992, containing 9.39 per cent, a variation from the mean of 3.38 per cent."

"The data show that the flours of this grade are placed upon our markets under very uniform conditions in respect of moisture This arises either from the fact that the quantity of moisture in the grains from which the flours are made is remarkably constant, or that the flours when prepared exhibit equal hygroscopic properties which tend to regulate the quantity of moisture therein contained. The only marked variations from the mean percentages of moisture are found in the two samples mentioned above and in No. 10834. Leaving out of consideration these three samples, the remarkable uniformity of moisture is made more strikingly apparent."

Following the suggestion that is made to omit the three extreme samples,

No. 12992 with 9.39% moisture
No. 12549 with 15.30% moisture
No. 10834 with 14.06% moisture

It is interesting to observe some of the results recorded for flours of the "best high-grade patent wheat flours on our markets" as recorded on page 1254 and of Class 11 designated as flours "having a composition not very greatly different from the high-grade patent flours already mentioned."

No. 10841 with 13.68% moisture
No. 10843 with 13.69% moisture
No. 10862 with 13.93% moisture

No. 11898 with 13.62% moisture
No. 10822 with 13.71% moisture
No. 10833 with 13.57% moisture
No. 12547 with 13.93% moisture

It is to be noted that these flours as well as half a dozen others that were tested are above 13½ per cent moisture content. In fact flours up to 13.93% moisture are given, and they are stated to have a remarkable uniformity of moisture. There is no suggestion of any unusual content of moisture until a sample with 14.06 is reached.

As previously noted these results are all on open air basis of drying.

24. Flours with 15 per Cent Moisture not considered Adulterated.

The Bureau of Chemistry regard from 9 to 15 as the normal range for the moisture content of flour. In the 14th Convention (1897) of the A. O. A. C. Dr. Bigelow of the Bureau of Chemistry, as Referee on Food Adulteration, in discussing the adulteration of flour says: "Its water content may be too high and cause it to be adulterated." He gives a table showing the range of composition for first class flour samples (page 117):

	Maximum	Minimum
Moisture	15.	9.
Ash	.8	.3
Proteids	15.	8.
Fiber	1.	.1
Ether Ext.	2.	.5
N free Ext.	90.	82.

Figures are also given for various "cereal flours and meals." Dr. Bigelow, who did many of the flour analyses reported in Bulletin 13, according to the prefatory note, says: "It must be understood that this table is only provisional, and it will frequently happen that products which are not adulterated will not fall within these limits. At the same time it is believed all samples of first class flour and meal should do so."

It is quite evident that the Bureau of Chemistry regarded 9 to 15 per cent as normal ranges of moisture in flour samples found on the market.

The American flours tested by the Bureau of Chemistry have

from time to time been highly commended because of their freedom from adulteration. Bulletin No. 63, page 10 says:

> "Flour. Owing to the firm attitude taken by American millers, the adulteration of staple brands of flour is practically unknown.***"

The flours today contain no more water than when this statement was made.

It has been suggested that when flour absorbs moisture in excess of 13½%, spoilage occurs. Fourteen and one-half vacuum standard is only about 13½ water oven standard; hence the spoilage point on the vacuum basis would be, above 14½. Spoilage may take place independent of any specific moisture content, depending upon the conditions of storage, germ content of flour, and condition of the wheat when milled. When spoilage does occur the Food and Drugs Act can be invoked independent of moisture content.

Corn meal has a 14% moisture standard and it is well known that corn meal has poorer keeping qualities than white flour. If the standards reflect keeping qualities then corn meal should have less, and not more, moisture than flour.

25. Moisture Content of Foreign Flours.

Foreign flours contain more water than American flours. This point was brought out at the Washington Hearing in 1915. Data upon this point are also recorded in Bulletin No. 13, page 1267, relative to the composition of typical French flours. These type samples contained respectively 15.42, 14.92, 15.58 and 14.74 per cent moisture. Other than cellulose and sucrose, it is stated: "In respect of the other constituents it may be said that the percentages obtained agree quite well with the results of the general analyses which have been conducted in this division." Page 1269.

The American Miller does not desire nor ask for a high moisture standard. His desire is to have 13½ per cent moisture mean the same now as it did when the standard was formulated. Then wheat and flour moisture methods remain on a parity.

26. Vacuum and Hydrogen Drying not Followed in Official Laboratories.

At the time the 13½ per cent moisture standard was formulated, comparatively few of the official laboratories of the United States dried food products in any other way than with the water or air bath.

W. H. Krug, Referee on Cattle Feeds in 1901, states on page 46, "The official methods for analysis of foods direct that the material shall be dried for five hours, at the temperature of boiling water in a current of dry hydrogen, or in vacuo. Neither of these methods has been generally adopted, as most laboratories are not equipped with the necessary facilities and are thus forced to use some other method." Frequently the Referee directed the analyst to determine moisture by the methods given in Bulletin 46 "or according to the methods used in your laboratory stating the method employed." Such directions were continued down to the report given in the Journal, A. O. A. C. for August 15, 1920.

Drying at the temperature of boiling water was for years the standard and common method in use for determining moisture in all foods. A good illustration is given in the Seventh Convention, 1890 Report, page 56, in discussing butter.

"Mr. Lupton asked whether the temperature was stated in the recommendation.

"Mr. Wiley said that it was; the temperature was fixed at the boiling point of water, the standard temperature adopted by the Association. Comparing Colorado with Louisiana, the difference in temperature would be very considerable, but the rate at which water went off from a substance depended generally upon the pressure and the temperature, and boiling water gave a constant condition everywhere, so with that as a standard all worked under the same conditions although there was a difference in actual temperature."

In 1902 Dr. Bigelow of the Bureau of Chemistry (Bulletin 66, page 11) states:

> "The drying of the samples in vacuo was not considered practicable, as few laboratories are equipped to use the method, and the large bulk of work also prohibited it."

In the 1907 Proceedings 53 analysts give moisture tests of portions of the same sealed sample sent out for purposes of testing. Five analysts used the vacuum method, only one above 75° C or so, eight used hydrogen drying, and the remainder, 40 out of the 53, used either the water or air bath or failed to report the method they used. Evidently some did not wish to go on record as to the method used.

Neither vacuum nor hydrogen drying represented methods in

general use in official laboratories preceding the time the 13½ per cent flour moisture standard was formulated or at the time the Food and Drugs Act was passed. During all these years trade practice has been based on water oven and air oven methods of drying.

27. No Short Weighing of Flour or Excess Moisture should be Entertained.

Suggestions for continuing the moisture methods used for establishing the 13½ per cent flour standard cannot be construed as suggesting that any short weighing in filling flour packages is to be entertained. All flours should be packed full weight, net 196 pounds per barrel, except when state laws require a different weight. In arriving at the net weight declarations of packages, in no case is more than 13½ per cent moisture to be allowed as determined by the open air methods in use and approved by the Division of Chemistry U. S. Department of Agriculture at the time the data for this 13½ per cent moisture standard was made.

This is the essence of the 13½ per cent standard as understood by the millers at the 1915 Hearing and as expressed by a number of millers as the record of the minutes of the Hearing show.

From Page 126 of Record of Hearing:

> Mr. Moses: We have various wheats. I don't think any miller should be permitted to pack less than 196 pounds to the barrel.
>
> Dr. Alsberg: Well, of course, we are not concerned with the miller's contract. We are concerned with this phase of the thing: That the law says that food in package form shall bear upon the outside of the package a plain conspicuous statement of the quantity of contents. How that statement shall be made is the only question we are considering.

From Page 125, condensed from a statement of Mr. Lingham.**** "Some of us millers realize that you are trying to formulate some definite basis of arriving at the actual flour content of packages. We believe that some basis of moisture must be adopted. I am speaking for myself more particularly. ******and for myself I believe that 13½ per cent moisture content would be accepted as a moisture content basis and all weights figured and determinations from that basis up or down."

The parts omitted relate to low moisture content flours which are now considered: As to the miller who might mill flour with 11 per cent moisture content, that phase of the question was fully discussed and was summarized by Mr. Crocker in reply to a hypothetical case of milling wheat with less than 7 per cent moisture propounded by Dr. Alsberg.

> "Mr. Crocker: Assuming that this miller could turn out a flour with 11 per cent moisture content, on what basis is he supposed to grind that flour? If the wheat he buys is correspondingly lower he should put in 196 pounds. If he is turning out flour with 10 or 11 per cent moisture content the assumption is that he is buying his wheat correspondingly. Really he is on the same basis with the other miller.

Briefly, the miller who mills his flour with 11 per cent moisture and buys his wheat with 7 to 9 per cent moisture, has no claim for packing a barrel of flour to weigh 191 or 192 pounds. The miller who buys wheat with 15 per cent moisture and is compelled to pack his flour with 13½ per cent moisture has a greater moral claim for packing flour with 15 per cent moisture than the miller who buys wheat with 7 per cent moisture and leaves 11 per cent in his flour. But this is all an academic discussion and fails to represent present commercial conditions. The way wheats are now graded and handled, if the dry wheats have the requisite quality they invariably command a premium in proportion to their dryness, and the extreme wet wheat sells at a lower price, as discussed in paragraph 3 of this article.

There is no alchemy in milling. There are over 7000 commercial mills in the United States capable of making, in about 100 days, flour that will last this country one year. There is no way a miller can get something for nothing and make a profit. As soon as a sound dry strong wheat is found it always commands a good stiff premium. Food Officials do not realize the keen competition that exists in the milling trade and the impossiblility of any miller putting out adulterated (with water) or a misbranded (short weight) flour and being able to "get by" or sell such an article to his trade. The flour trade is too sensitive and competition too keen to permit such practices being carried to any appreciable extent and the Millers National Federation and other trade organizations would soon expose any such practices.

28. Results of the First Co-operative Tests A. O. A. C. with Drying Flour in Vacuum Oven and in Water Oven.

In a preceding paragraph it is mentioned that in 1910 the A. O. A. C. directed that a study be made of the methods for testing cereal products. Associate Referee White of North Dakota made such studies and reported the following results in 1911. These results are interesting to note.

"RESULTS OF ANALYSIS.—MOISTURE.

Comparative results obtained by different methods for determining moisture in flour.

	Sample A (Fife)			Sample B (Durum)		
	Method A (Water Oven)	Method B (Vacuum)	Method C (Basset's)	Method A (Water Oven)	Method B (Vacuum)	Method C (Basset's)
	Percent	Percent	Percent	Percent	Percent	Percent
Lelia Dunton, Manhattan, Kan.	11.01	11.90	11.55	10.52	11.365	11.145
B. R. Jacobs, Washington, D.C.		[1]11.72	....	.....	[1]11.12	.....
Agricultural College, N. D.	10.13	.....	10.07	9.54	.....	9.65
R. F. Beard, Agricultural College, N. D.	(10.32) ([3]10.91)	[2]11.88	10.09	(9.94 11.64) ([3]10.91 [2]11.47)		9.88 ....
C. K. Glycart, St. Paul, Minn.	10.24	11.89	9.97	9.55	11.06	9.35
H. L. White, Agricultural College, N. D.	(10.50) (10.18)	10.56	10.595	(10.43) (9.65)	[2]10.42	10.53 .....
G. A. Olson, Pullman, Wash.	[4]7.49	.....		[4]6.34	.....	
Averages.....	10.47	11.59	10.455	10.076	11.18	10.109

1. "Drying in a vacuum 4½ hours."
2. In vacuum oven at 70° C for periods ranging from 24 to 144 hours.
3. In aluminum dishes fitted with covers.
4. Kept in water oven at boiling temperature for 3 hours; not included in average.

Miss Leila Dunton says: "Moisture Method A we do not use in this laboratory for flour samples as it fails to remove all moisture. Method B is very good. The vacuum desiccator we find very convenient and accurate."

Associate Referee White says in his report to the Association: "These results strengthen the conviction that for accurate results, when working with materials that are affected by the temperature of boiling water, the method utilizing the vacuum oven or desiccator is the most accurate; but this method should be standardized as regards temperature and other conditions. As indicated in the footnotes to the preceding table, the time varied from 4½ hours to 144 hours, and the temperature from room temperature to 70° C."

"Method C (Bassett's) may well be considered further as a quick method for the approximate determination of both moisture and fat."

It is to be noted that vacuum drying gave results about one per cent higher (1.12) than drying in the water oven. All of these tests show a low per cent of moisture. The flour was milled in the experimental mill of the North Dakota Experiment station. Sample A, is designated a straight flour and sample B, a patent flour from durum wheat. Tests reported show .70% (ash) for sample A and the same for sample B (.697). If the flours were milled so as to show a low moisture, the high ash could easily have resulted from lack of proper tempering of the wheat before milling, as high ash results from such a condition.

The Bassett method was one used extensively by the North Dakota station in flour investigations in connection with weight variations of flour during storage. This method, as is to be noted, gives the same results as obtained by direct drying in the water oven.

Omitting the extreme result, the five chemists using the vacuum method of drying obtained results that differed by .58% (Sample B, 11.64 high, 11.06 low.) One analyst however, Mr. H. J. White, the Associate Referee, obtained from .76 to 1.01 per cent below the average by the vacuum method. This variation cannot be considered as much the fault of the analyst as due to defects of the method. In any series of flour moisture tests similar variations may occur.

29. Recent A. O. A. C. Work on Flour Moisture.

For the past few years the Association has studied vacuum drying without heat compared with vacuum oven drying at 70° C, and also at 100° C with 20 to 28 inches of vacuum. The results fail to check. For example one analyst from the Bureau of Chemistry reports maximum moisture by drying in a vacuum desiccator over sulphuric acid without heat for several days while another analyst of the

Bureau reports maximum results by drying in a vacuum oven at 100° C with 27 inches of vacuum. Then again, different desiccating agents give different results. In some of the tests it is even reported that the flour was dried in an 8 inch Hempel desiccator, containing a litre of sulphuric acid. (J. O. A. A. C. Aug. 15, 1915, page 196)

In the case of sulphuric acid as a desiccating reagent, Associate Referee Clarke mentions the necessity of avoiding the use of any discolored sulphuric acid, and he gives the treatment necessary in order to remove traces of organic matter from the acid. "Avoid the use of discolored acid, as it frequently gives off some fumes of sulphur dioxide." J. O. A. A. C. Aug. 15, 1920 page 49.

You can start a test with c. p. sulphuric acid but when you apply suction to the desiccator fine flour particles are liable to be drawn from the dishes and be deposited in the acid causing it to give off sulphur dioxide.

Sulphur dioxide, even in traces, has a marked action upon wheat gluten proteins. When flour is fumigated with sulphur even lightly as in case of fumigating for contagious diseases, such flour is rendered practically valueless for bread making. Sulphur dioxide in mere traces causes a catalytic action that splits the wheat protein molecules. The fact that moisture results obtained by drying over sulphuric acid in a vacuum desiccator are so erratic, sometimes more and sometimes less than drying in the vacuum oven with extreme heat and vacuum conditions, may be due to this catalytic action of sulphur dioxide, occurring in some but not all tests. Carefully conducted microscopic tests show that such changes occur.

Proteins of most foods are not as sensitive to the action of sulphur dioxide as wheat proteins, and because wheat proteins behave in this way does not invalidate the use of vacuum desiccating drying for foods in general.

In vacuum drying without heat, great care is necessary in the use of the dehydrating agent. Flour is so exceeding fine it is easily stirred, and too much shaking of the desiccator, as directed to mix the acid, could easily raise a little flour dust that would be deposited in the acid and make sulphur dioxide.

Associate Referee Clark's Report on Water in Foods and Feeding Stuffs appearing in the Journal of the Association of Official Agricultural Chemists Feb. 15, 1921, is particularly worthy of note and the following quotations are from this report.

"In food control work a number of different procedures are used for the determination of moisture or water, the method employed depending on the nature of the material and equipment of the laboratory. These methods can be classified as heating, desiccator, refractometer, densimetric, or other procedures. The temperature, pressure, type of apparatus, and other factors differ in each class. It is not surprising, therefore, that the determination of water is quite variable and that it is next to impossible for two laboratories to obtain good checks.

"As ordinarily determined, the water or moisture in food products is derived from several sources, the principal ones being loosely bound moisture, water of constitution, and water derived from the decomposition of organic materials. If the moisture is determined by difference, low boiling and gaseous substances other than water will be lost. In the process of drying, other changes, more or less pronounced, always take place. Certain constituents may be oxidized, as in linseed meal, or other substances containing a drying oil. Many feed materials lose weight on heating until a certain minimum weight is obtained, when they begin to increase in weight.

"As ordinarily used, the term 'per cent of moisture,' or 'per cent of water' in a food material is intended to mean the loosely bound water in the material, or, in substances in which the water occurs as a solvent, sirups, etc., the actual uncombined water present. It is not possible in ordinary work to determine this water without including water from other sources or other volatile materials. For the purpose of this association a few well-selected methods are needed which will give a consistent measure of the moisture in such substances as feed materials, and a measure of the actual water in such substances as sirup. Such selected methods should give comparative results when used with care with the equipment found in the ordinary laboratory.

"It is quite possible that the treatment in vacuo at the temperature of boiling water is too strenuous to remove the moisture from some materials without other changes being too pronounced. It may be a better procedure to dry such material at a lower temperature in vacuo. The statement 'in a current of dry hydrogen or in vacuo' is extremely vague, and it is not probable that concordant results could be obtained if these instructions were followed. The simple specification 'in vacuo' may mean less than 1 mm., or 70 mm. or absolute pressure." Page 344.

Last year (1921) the A. O. A. C. decided (2) "That work on the determination of moisture and ash be discontinued until further research develop more desirable methods." (J.A.O.A.C. Vol. VI No. 2)

30. Results of Later Comparative Moisture Tests.

Mr. L. E. Leatherock reports a series of co-operative tests conducted by 69 chemists all reporting on the same sealed sample of flour sent out for the purpose of testing methods employed in different laboratories. (See July 1922 Journal of the American Association of Cereal Chemists, pages 102-107.)

> 59 laboratories used air ovens for drying the flour at about 103° to 105°C for periods ranging from 4 to 8 hours. General average moisture 14.15 per cent.
>
> 10 laboratories used vacuum ovens, with temperatures ranging from 95 to 140°C with time limits from 5 minutes to 5 hours, and vacuum employed not reported. General average moisture 14.45 per cent, ranging from 14.1 to 14.74.

No tests are reported with water oven drying. Had such tests been made they would have shown appreciably lower results than air drying at 103-5, presumably .6 per cent.

> "About the only definite conclusion we are able to draw from all this data was the fact that we did not know how much moisture the flour contained, and that the layman unfamiliar with laboratory work would be fully justified in wondering why more concordant results were not obtained on check samples that are now frequently sent around in so-called air-tight containers."

Among the conclusions drawn are: "Laboratories using air ovens and vacuum ovens cannot check between one another; vacuum oven gives higher results." The necessity for standardizing time and temperature, and the limitation of the range of temperature for air ovens is noted.

Mr. Leatherock's report is of particular interest as it shows that in commercial practice over 85% of the laboratories that do regular flour testing use air drying, and 15 per cent use vacuum oven drying.

Vacuum drying does not represent present day trade conditions. The expense of installing a vacuum oven in many laboratories would be prohibitive. The statement made by the Bureau of Chemistry in 1902 that "the drying of samples in vacuo was not considered practicable as few laboratories are equipped to use this method, and the large bulk of work also prohibits it," is particularly applicable to flour mill control work today.

31. Moisture Content of Flours Determined by Different Methods.

Shutt and Moloney in Trans. of the R. Sc. of Can., 1917, give some interesting facts on this point.

They first tested drying flour in dry hydrogen, and then in vacuum at 100° C, 29½ inches vacuum. The vacuum drying gave a suggestion higher results—ranging from .05 to .11 per cent, subsequent drying in air following hydrogen drying gave no change. The following table presents results obtained in various ways of drying:

Comparison of results from vacuum oven drying with those from electric air oven at 100°C and at 110° C., and water oven at 91° C.

Series	Oven	Temperature	Time	Percentage of moisture a	b	c	d
A.....	Vacuum	100°C	5	12.63	12.64	12.60	.....
	Vacuum	100°C	+ 5	12.79	12.77	12.77	.....
B.....	Vacuum	100°C	5	12.52	12.55	12.53	.....
	El. air	100°C	+16	11.42	11.57	11.67	.....
	Vacuum	100°C	+ 5	12.57	12.58	12.64	.....
C.....	Vacuum	100°C	5	12.44	12.42	12.43	12.46
	El. Air	110°C	+17	12.25	12.26	12.26	12.29
	Vacuum	100°C	+ 5	12.55	12.59	12.59	12.60
D.....	Vacuum	100°C	5	13.43	13.41	....	.....
	Water Oven	91°C	+18	11.92	11.97	.....	.....
	Vacuum	100°C	+ 5	13.42	13.38	.....	

Four series of tests were made, A, B, C and D, and triplicate samples a, b, c, d, tested in each series. For series A, it is noted under time, 5 hours, that the moisture results for vacuum drying are 12.63 12.64, 12.60. Then these same samples were dried 5 hours longer, and 12.79, 12.77 and 12.77% moisture was secured, a gain of .15% resulting from five hours additional drying. In series B, samples were first dried 5 hours in the vacuum oven at 100°C, then these same samples were dried 16 hours additional in an air oven at 100° C, and then dried again five hours in the vacuum oven. It is to be noted that the air drying showed less moisture but that .15 more moisture was obtained after five hours additional vacuum drying. The drying at 110° C air maintained the weights more closely with vacuum drying than any other method. Water oven drying gave about 1.50 less moisture than vacuum drying and air oven at 100° C about one per cent less than vacuum drying.

An examination of Shutt and Moloney's results show that with

the vacuum drying prolonged beyond five hours, there was a general tendency for higher moisture results. This additional loss could have come from mechanical losses caused by the suction of the air pump removing fine flour particles. As high as .17% mechanical losses may have occurred during each 5 hours' vacuum drying.

32. Standards of Purity for Food Products.

The Food and Drugs Act makes no provision for Food Standards. The Secretary of Agriculture, in 1920 reported. (Page 60)

"In order to secure the more effective and efficient enforcement of the food and drugs act, the department should be specifically authorized to establish standards of strength, quality and purity for the articles subject to its provisions, and ample power should be given it to enforce compliance with the standards." While the standards do not have the force of law, nevertheless reasonable standards, founded upon the best trade practices, are alike helpful in the enforcement of the Food and Drugs Act and in commercial transactions.

In the appropriation act for the Department of Agriculture, 1902, Congress authorized the Secretary of Agriculture to make certain investigations relative to foods, and among the specifications are: "To enable the Secretary of Agriculture, in collaboration with the Association of Official Agricultural Chemists, and such other experts as he may deem necessary, to establish standards of purity for food products, and to determine what are regarded as adulterations therein, for the guidance of the officials of the various states and of the courts of justice."

One set of standards—Circular No. 10—was issued under this act. No standards for flour or grain products are included in this set of standards proclaimed November 20, 1903.

The following session of Congress passed the same act but ending with the words: "adulteration therein." The appropriations act for 1903-1905 did not carry the clause reading: "for the guidance of officials of the various states and of the courts of justice." Hence the Standards Committee in their work, conducted under the later appropriations act, 1903-1905, did not formulate standards for the guidance of officials and courts. When the Standards Committee submitted its report containing the definition of flour and the 13½ per cent moisture standard to Secretary Wilson, December 19, 1904, the Committee supposed they were working under the act giving authority for formulation of standards for the guidance of officials

and courts. (See page 10 Bureau of Chemistry Bulletin 69 Revised part 1.) Circular 19, issued later, superseded the standards of December, 1904, circulars 13 and 17, and also circular 10 which is the only standard formulated and published for the guidance of officials and courts. Circular 136, Standards of Purity for Food Products, suspends circulars 13, 17 and 19, but does not mention circular 10. It is generally supposed that all the standards formulated by the Standards Committee in its early work, prior to the Food and Drugs Act, was "for the guidance of officials and courts," but the acts of Congress show that this is not the case.

33. The Moisture Standard Should not be Changed by Changing the Method of Determining Moisture.

The 13½ per cent moisture standard should have a definite and fixed meaning. It should mean the same today as when first promulgated and as understood by the millers at the 1915 Hearing. Three courses are open for accomplishing this:

1. Continue the open air moisture drying methods with time and temperature limits as in former A. O. A. C. methods for foods and as is now official for sugar, milk, butter, cheese and many agricultural products;

2. Use "vacuo" drying with heat, specifying the time, temperature and vacuum and allowing for the fine flour removed during the drying process. This would necessitate a moisture standard of 14½ per cent or more and would include water of hydration. About fourteen and one half per cent moisture under such conditions corresponds with the standard formulated in 1904;

3. Adopt a definition for flour without reference to moisture content and then the U.S. Department of Agriculture issue a service and regulatory announcement fixing 13½ per cent moisture, water oven basis or 14½ per cent, vacuum oven basis, as the maximum allowable for adjusting the weight of flour packages.

The introduction and development of moisture methods that give one or more per cent moisture than was obtained by the methods used for securing the data upon which the standard was formulated simply reduce the moisture standard for flour one per cent or more.

This unintentional changing of the standard seriously affects flour milling operations and the transportation, sale and legal status of flour. The whole matter should receive careful consideration and proper adjustment.

Regulation 4, Food and Drugs Act specifies that the methods of the A. O. A. C. shall be used as official methods in testing food, provided, however, that any method satisfactory to the Bureau of Chemistry may be employed. Under the provisions of this regulation the Bureau of Chemistry and then the Department of Agriculture must act when methods of analysis and standards fail to harmonize and fail to reflect the best trade practices.

It is not surprising as methods of analysis change, that they affect old standards founded upon former methods, and that conditions such as exist in relation to the method for determining moisture in flour and the moisture standard should occur. The wonder is that more such anomalous conditions do not occur.

34. The Relation of the Standard to the Food and Drugs Act.

A moisture standard for flour, it would seem, should harmonize with the Food and Drugs Act and the Authorized Regulations. As the present standard was formulated two years prior to the advent of the act, the Standards Committee could not anticipate the law, the standard, however, can be interpreted to harmonize with the law. A moisture standard cannot consistently maintain that a sound flour at no time and under no conditions, (that is, without regard to any other factor) shall not exceed 13½ per cent moisture. We know that flour milled with less than 13½ per cent moisture will, under natural conditions absorb more than 13½ per cent, and still be sound. The Food and Drugs Act and Regulation 26 take into account such natural weight deviations due to either absorption or evaporation of moisture. No standard should attempt to limit what the law and regulations permit. Furthermore, hygroscopicity follows nature's laws. The water added by nature cannot be regarded as water added by the miller. Provided the flour contains its full original dry matter, loss or gain of moisture in no way affects the consumer. To require the drying of flours that are sound and originally packed full weight without excess moisture, benefits no one except, perhaps those who want to dry the flour or the persons who want to breach a contract. Drying may result in injury, as flour is easily affected by heat. A standard should not be invoked to do unreasonable or unnecessary acts which are not in harmony with the law, authorized regulations, or good commercial practice.

35. Moisture Absorbed or Lost During Manufacture.

There is another phase of this question that must be taken into

consideration in handling flour. A manufacturing tolerance is necessary to cover variations in weight due to changes in humidity of the atmosphere while the flour is being manufactured and packed.

The original Regulation No. 29, Statement of Weight or Measure, suggests such a tolerance. It reads:

> "(b) A reasonable variation from the stated weight for individual packages is permissible, provided this variation is as often above as below the weight or volume stated. This variation shall be determined by the inspector from the changes in humidity of the atmosphere from the exposure of the package to evaporation or to absorption of water and the reasonable variations which attend the filling and weighing or measuring of a package."

The influence of humidity upon the moisture content and weight of flour during milling has been studied by the U. S. Department of Agriculture. See Bulletin No. 1013. Humidity conditions in the mill often change suddenly and cannot be foretold by the miller, as normal humidity conditions cannot be uniformly maintained in a flour mill.

In the bulletin cited it is stated that when hard wheats of various moisture contents were tempered to the same moisture basis of 15 per cent and were milled under different humidity conditions:

> "No very pronounced relation between the moisture content of the flour and that of the wheat before tempering was shown, or in other words, no relation was shown of moisture content of flour to the quantity of temper water added to the wheat. On the other hand, a decided tendency was shown for the moisture content of the flour to increase as the relative humidity increased, a difference of 10 per cent in relative humidity making an average difference of approximately one half of one per cent in the moisture content of the flour."

These changes in the humidity affecting the moisture of the flour as determined by the U. S. Department of Agriculture, are reasonable variations which attended the manufacture of flour and the filling and weighing of flour packages. It is believed that such weight variations should constitute a tolerance occurring in milling and packing conducted in compliance with good commercial practices. Regulation No. 26 does not appear to either exclude or include such a tolerance. It is an uncontrollable variation that occurs and affects the weight as often in one direction as it does in another.

The operative miller must have a margin to cover such moisture changes occuring in milling operations. The best trade practice should ultimately be taken as a guide and the U. S. Department bulletin cited is comparable with the best trade practices as to influences of humidity changes in flour milling.

36. Packing and Weighing Flour to Comply with Food and Drugs Act.

Flour packers are generally experienced and high grade men, and would resent a suggestion of short weighing on the part of an employer; and a miller could not deliberately short weigh his flour without the knowledge of the packer. The weigh masters of most states and cities frequently inspect the miller's scales. The railroads and transportation companies also check the mill weights. If the miller did not deliver accurate weights his books would not balance, the miller's bankers would know it and in turn would refuse to give him credit. Errors may arise in filling and weighing packages but actual intentional short weighing of flour is of unusual occurrence. When it does occur it is inexcusable.

Some means of weighing and inspection should be developed whereby the miller, the inspector, and the flour dealer can alike unite upon a system that will secure reasonable accuracy. High grade flour packers, might be appointed accredited weighmasters after complying with certain state or national requirements. The miller should have some way whereby he can pack his products, have them inspected and passed and not be placed in the embarrassing position of being libeled because of uncontrollable atmospheric conditions.

The purchaser is entitled to flour packed full weight without excess of moisture. To determine if this has been done, full consideration must be given to all the factors mentioned in section 7 and discussed in sections 8 to 12. The great difficulty is lack of appreciation of the extent to which flour, packed so as to fully comply with the Food and Drugs Act, may subsequently vary in weight and moisture content while the dry matter weight remains practically unchanged and the value of the flour is unimpaired.

37. Principles Involved in Determining Flour Moisture and Weight as Indorsed by Trade Organizations.

A committee consisting of members from the Millers National

Federation, The Association of Operative Millers, and the American Association of Cereal Chemists, known as the Allied Associations' Committee has considered this question of weight and moisture content of flour. They recommended that only the "free moisture" of flour be recognized as moisture and not other forms, or mechanical losses due to aspiration; and also that weight regulations be based on not to exceed 13½ per cent moisture as determined by the methods used in formulating the standard.

These resolutions provide for the continuance of the 13½ per cent moisture standard on its original basis, and emphasize that a moisture standard rests upon the method used for the determination of the moisture. One should not be changed without a corresponding change of the other.

38. Adjustment.

This whole question of the weight and moisture content of both wheat and flour and their maintenance on a parity basis rests upon a few fundamental facts as stated. A common method for determining moisture in each has been used, and a wheat standard and a flour standard have alike been formulated on that basis (water oven drying). The grain moisture method is provided for in the Grain Standardization Act, and the Secretary of Agriculture is authorized to establish the method. He is also chief executive officer of the Food and Drugs Act, and it would seem that he is the one to determine whether the moisture method for wheat and flour shall remain on a parity and the 13½ per cent flour moisture standard be continued as originally proclaimed, or whether by the use of another and different method for determining moisture in flour, the parity between wheat and flour shall be changed and the present flour moisture standard lowered.

39. Economic Features Involved.

There are certain industrial and economic features involved in a flour moisture standard that need to be noted. Dr. Alonzo Taylor in "War Bread," page 76 says: "In comparing American and European extractions, the water content of flours must be kept in mind. Here the flour contains about 13 per cent of water, in Europe higher water content is permitted, 17 per cent being common." American flour must compete with European flour. European governments without exception encourage the importation of wheat so their own

millers can manufacture flour. European governments all want to import raw material, as wheat, and make manufactured products, and hence try to avoid importing any flour. The American Miller does not want a high moisture standard. He has advocated just as low a standard as he can consistently operate under, 13½ per cent. But to change the method of determining the moisture so as to lower the standard to 12½ per cent, places him in a position that he cannot successfully mill flour and compete in the world's markets. To attempt to operate on a 12½ per cent standard would raise the price of flour and also result in still further curtailment of export flour trade. Furthermore, if the manufacture of flour is curtailed by standards, and the domestic demand for wheat is lessened the farm price of wheat drops correspondingly.

If an agitation is started in this country to the effect that the American Miller is adulterating his flour with water some foreign government may have a pretense of an excuse for excluding American flour. which treaty rights now prevent. Some years ago an European country tried to exclude American flour claiming it was adulterated with copper sulphate, and it took time and costly effort to disprove the libel.

During present reconstruction times it would be most unfortunate to suggest that the American Miller adulterates his flour with water, unless the facts actually warranted such statements, because it would be another factor tending to create unrest and distrust.

Export and domestic flour business are done on narrow margins and to change an established weight standard one per cent, by changing a moisture method, would be a deciding factor as to whether a mill be operating or idle. And when flour mills are not operating the demand for wheat lessens and the price drops. I do not believe that any one desirous of broadening American markets, would knowingly take a step that would unquestionably lower the farm price of wheat. The world's commercial relations are so delicately poised today that it takes but little to disturb trade conditions.

The American Miller justly feels proud of his product and has never countenanced adulteration in any form. Washington at his mill in Mount Vernon set a very high standard, his flour being accepted in many of the markets of the world without inspection. Later Abraham Lincoln honored the craft as a grist mill hand and was known as "Honest Abe". Long before the Food and Drugs Act the millers were instrumental in having a special law passed

preventing the adulteration of flour—the mixed flour law, which safeguards the purity of flour more effectually than the Food and Drugs Act. Various attempts have been made to repeal the mixed flour law, but so far these attempts have been unsuccessful. During the world war when the question of bread supply was so acute and promised to be one of the features that meant success to the Allied Army, it was the American millers under the guidance of Herbert Hoover that developed and put into execution the plans for making and distributing flour. They discharged this trust in a most honorable way. The Millers National Federation and other trade organizations representing the best trade practices have made their recommendations as to the basic principles that are involved in this question of the moisture content and weight of flour, and these recommendations are in harmony with the Food and Drugs Act, the Authorized Regulations and the best trade practices.

INDEX TO TOPICS DISCUSSED

(Numbers Refer to Paragraph Sections.)